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Methyl (4-oxo-1-phenyl-4,5-dihydro-1*H*-pyrazolo[3,4-*d*]pyrimidin-5-yl)acetate

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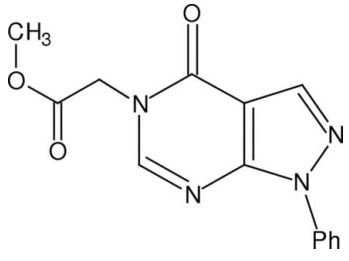
Key indicators: single-crystal X-ray study; T = 173 K; mean σ(C–C) = 0.002 Å; R factor = 0.035; wR factor = 0.096; data-to-parameter ratio = 15.2.

The title compound, $C_{14}H_{12}N_4O_3$, a pyrazolopyrimidine derivative, displays normal geometrical parameters. The dihedral angle between the mean planes of the pyrazolopyrimidine unit and the phenyl ring is 26.14 (4)°. The non-H atoms of the ester side chain are coplanar (r.m.s. deviation = 0.009 Å) and this plane is almost perpendicular [dihedral angle = 84.31 (4)°] to the central ring system.

Related literature

For related structures, see: Wen *et al.* (2004); Oliveira-Campos *et al.* (2006) and Portilla *et al.* (2005). For background literature, see: Garg *et al.* (1990); El-Feky & Abd El-Samii (1996); Ismail, *et al.* (2003); Devesa, *et al.* (2004) and Russo *et al.* (1993).



Experimental

Crystal data

$C_{14}H_{12}N_4O_3$
M_r = 284.28
Triclinic, $P\overline{1}$
a = 6.8628 (8) Å
b = 7.6128 (9) Å
c = 12.4949 (16) Å
α = 87.036 (10)°
β = 89.608 (11)°
γ = 79.973 (8)°
V = 641.97 (13) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.11 mm^{-1}
T = 173 (2) K
0.45 × 0.43 × 0.42 mm

Data collection

Stoe IPDSII two-circle diffractometer
Absorption correction: none
6143 measured reflections
2914 independent reflections
2601 reflections with $I > 2\sigma(I)$
R_{int} = 0.022

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.035
$wR(F^2)$ = 0.096
S = 1.02
2914 reflections
192 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.28 e Å^{-3}
$\Delta\rho_{min}$ = −0.20 e Å^{-3}

Data collection: *X-AREA* (Stoe & Cie, 2001); cell refinement: *X-AREA*; data reduction: *X-AREA*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXL97*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB2372).

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supplementary materials

Acta Cryst. (2007). E**63**, o2566 [**doi:10.1107/S1600536807018934**]

Methyl (4-oxo-1-phenyl-4,5-dihydro-1*H*-pyrazolo[3,4-*d*]pyrimidin-5-yl)acetate

H. S. Yathirajan, S. Bindya, B. K. Sarojini, B. Narayana and M. Bolte

Comment

Pyrazolo[3,4-d]pyrimidines and their derivatives are of interest as potential bioactive molecules. Various pyrazolopyrimidine derivatives are reported to have antileishmanial, antihypertensive, antibacterial and antifungal, antiangiogenic, antiinflammatory and analgesic activities. The title compound, a new pyrazolopyrimidine derivative, (I), $C_{14}H_{12}N_4O_3$, has been synthesized and its crystal structure determined.

Experimental

4-Hydroxy-1-phenyl pyrazolo[3,4-d]pyrimidine (21.2 g, 0.1 mol) in 180 ml acetone was stirred with anhydrous potassium carbonate (16.5 g, 0.12 mol) at room temperature and methyl chloroacetate (12.2 g, 0.1 mol) was added to it drop-wise. The reaction mixture was then refluxed for 8 h. Progress of the reaction was monitored by TLC. The acetone was distilled out and the residue was diluted with 250 ml water. The solid obtained was filtered, washed with water and then recrystallized from methanol to obtain the title compound as colourless needles (Yield: 95%; m.p.: 421-424 K). Crystals of (I) suitable for X-ray diffraction were obtained from acetone by slow evaporation. Analysis for (I), $C_{14}H_{12}N_4O_3$: Found (Calculated): C: 59.01 (59.15); H: 4.11 (4.25); N: 19.62% (19.71%). ^{1}H-NMR (400 MHz, $CDCl_3$): δ 3.78 (s, 3H, -CH_3), 4.73 (s, 2H, -CH_2), 7.34 (t, 1H, ArH), 7.48 (t, 2H, ArH), 7.94 (s, 1H, ArH), 7.99 (d (J = 6.08), 1H, ArH), 8.23 (s, 1H, ArH).

Refinement

The H atoms were found in a difference map, repositioned in idealised locations (C—H = 0.95-0.99 Å) and refined as riding with U_{iso}(H) = 1.2U_{eq}(C) or U_{iso}(H) = 1.5U_{eq}(C_{methyl}). The methyl group was allowed to rotate but not to tip to best fit the electron density.

Figures

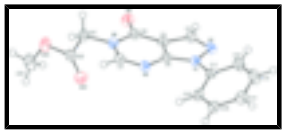


Fig. 1. Perspective view of (I) with the atom numbering; displacement ellipsoids are at the 50% probability level (arbitrary spheres for the H atoms).

Methyl (4-oxo-1-phenyl-4,5-dihydro-1*H*-pyrazolo[3,4-d]pyrimidin-5-yl)acetate

Crystal data

$C_{14}H_{12}N_4O_3$	$Z = 2$
$M_r = 284.28$	$F_{000} = 296$
Triclinic, $P\overline{1}$	$D_x = 1.471$ Mg m^{-3}

Hall symbol: -P 1	Mo $K\alpha$ radiation λ = 0.71073 Å
a = 6.8628 (8) Å	Cell parameters from 5143 reflections
b = 7.6128 (9) Å	θ = 3.9–26.8º
c = 12.4949 (16) Å	μ = 0.11 mm^{-1}
α = 87.036 (10)º	T = 173 (2) K
β = 89.608 (11)º	Block, colourless
γ = 79.973 (8)º	0.45 × 0.43 × 0.42 mm
V = 641.97 (13) Å^3	

Data collection

Stoe IPDSII two-circle diffractometer	2601 reflections with $I > 2\sigma(I)$
Radiation source: fine-focus sealed tube	R_{int} = 0.022
Monochromator: graphite	θ_{max} = 27.6º
T = 173(2) K	θ_{min} = 3.7º
ω scans	h = −8→8
Absorption correction: none	k = −9→9
6143 measured reflections	l = −16→13
2914 independent reflections	

Refinement

Refinement on F^2	H-atom parameters constrained
Least-squares matrix: full	$w = 1/[\sigma^2(F_o^2) + (0.0504P)^2 + 0.1857P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)]$ = 0.035	$(\Delta/\sigma)_{max} < 0.001$
$wR(F^2)$ = 0.096	$\Delta\rho_{max}$ = 0.28 e Å^{-3}
S = 1.02	$\Delta\rho_{min}$ = −0.20 e Å^{-3}
2914 reflections	Extinction correction: SHELXL97, $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
192 parameters	Extinction coefficient: 0.070 (7)
Primary atom site location: structure-invariant direct methods	
Secondary atom site location: difference Fourier map	
Hydrogen site location: inferred from neighbouring sites	

Special details

Experimental. ;

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted R-factor wR and goodness of fit S are based on F^2, conventional R-factors R are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > 2\text{sigma}(F^2)$ is used only for calculating R-factors(gt) etc. and is not relevant to the choice of reflections for refinement. R-factors based on F^2 are statistically about twice as large as those based on F, and R- factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
N1	0.39377 (13)	0.15427 (12)	0.60302 (7)	0.0202 (2)
N2	0.59660 (13)	0.14301 (13)	0.61111 (7)	0.0234 (2)
C3	0.65154 (16)	0.22729 (14)	0.52324 (9)	0.0228 (2)
H3	0.7840	0.2402	0.5073	0.027*
C4	0.48698 (15)	0.29526 (14)	0.45657 (8)	0.0207 (2)
C5	0.32439 (15)	0.24664 (14)	0.51033 (8)	0.0193 (2)
N6	0.13029 (13)	0.28745 (13)	0.47750 (7)	0.0228 (2)
C7	0.10838 (16)	0.38329 (15)	0.38733 (9)	0.0237 (2)
H7	−0.0225	0.4156	0.3599	0.028*
N8	0.25607 (14)	0.44203 (13)	0.32814 (7)	0.0233 (2)
C9	0.45997 (16)	0.40085 (14)	0.35729 (8)	0.0224 (2)
O9	0.58579 (13)	0.45707 (12)	0.30136 (7)	0.0298 (2)
C11	0.28917 (16)	0.08388 (14)	0.69005 (8)	0.0209 (2)
C12	0.37358 (19)	0.06459 (19)	0.79214 (10)	0.0332 (3)
H12	0.4986	0.0979	0.8036	0.040*
C13	0.2726 (2)	−0.0040 (2)	0.87694 (10)	0.0412 (3)
H13	0.3298	−0.0178	0.9465	0.049*
C14	0.0892 (2)	−0.05258 (17)	0.86134 (10)	0.0320 (3)
H14	0.0208	−0.0982	0.9199	0.038*
C15	0.00682 (17)	−0.03376 (15)	0.75916 (9)	0.0264 (2)
H15	−0.1185	−0.0668	0.7480	0.032*
C16	0.10647 (16)	0.03323 (15)	0.67283 (9)	0.0239 (2)
H16	0.0505	0.0443	0.6030	0.029*
C21	0.20561 (18)	0.55468 (15)	0.23041 (9)	0.0269 (2)
H21A	0.2833	0.6529	0.2277	0.032*
H21B	0.0636	0.6085	0.2319	0.032*
C22	0.24787 (16)	0.44799 (16)	0.13065 (9)	0.0241 (2)
O22	0.28503 (14)	0.28799 (12)	0.12993 (7)	0.0346 (2)
O23	0.23286 (13)	0.55953 (12)	0.04324 (6)	0.0327 (2)
C23	0.2652 (2)	0.4720 (2)	−0.05810 (10)	0.0403 (3)
H23A	0.3964	0.3966	−0.0576	0.061*
H23B	0.2571	0.5626	−0.1173	0.061*
H23C	0.1638	0.3980	−0.0676	0.061*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0177 (4)	0.0235 (4)	0.0201 (4)	−0.0055 (3)	0.0006 (3)	0.0001 (3)
N2	0.0173 (4)	0.0279 (5)	0.0256 (5)	−0.0057 (3)	0.0007 (3)	−0.0013 (4)

C3	0.0193 (5)	0.0253 (5)	0.0250 (5)	−0.0063 (4)	0.0032 (4)	−0.0035 (4)
C4	0.0207 (5)	0.0222 (5)	0.0207 (5)	−0.0070 (4)	0.0042 (4)	−0.0035 (4)
C5	0.0203 (5)	0.0203 (5)	0.0183 (5)	−0.0060 (4)	0.0023 (4)	−0.0027 (4)
N6	0.0199 (4)	0.0281 (5)	0.0210 (4)	−0.0061 (3)	0.0003 (3)	0.0002 (3)
C7	0.0220 (5)	0.0282 (5)	0.0215 (5)	−0.0061 (4)	0.0005 (4)	−0.0012 (4)
N8	0.0272 (5)	0.0262 (5)	0.0172 (4)	−0.0068 (4)	0.0014 (3)	0.0007 (3)
C9	0.0254 (5)	0.0234 (5)	0.0198 (5)	−0.0073 (4)	0.0049 (4)	−0.0043 (4)
O9	0.0311 (4)	0.0340 (5)	0.0262 (4)	−0.0118 (3)	0.0097 (3)	0.0009 (3)
C11	0.0229 (5)	0.0205 (5)	0.0194 (5)	−0.0048 (4)	0.0030 (4)	−0.0001 (4)
C12	0.0319 (6)	0.0483 (7)	0.0231 (6)	−0.0183 (5)	−0.0025 (5)	0.0032 (5)
C13	0.0460 (8)	0.0622 (9)	0.0196 (6)	−0.0232 (7)	−0.0031 (5)	0.0061 (6)
C14	0.0373 (7)	0.0366 (6)	0.0238 (6)	−0.0125 (5)	0.0074 (5)	0.0031 (5)
C15	0.0248 (5)	0.0268 (6)	0.0283 (6)	−0.0074 (4)	0.0046 (4)	0.0014 (4)
C16	0.0235 (5)	0.0268 (5)	0.0218 (5)	−0.0063 (4)	0.0004 (4)	0.0011 (4)
C21	0.0340 (6)	0.0269 (5)	0.0196 (5)	−0.0062 (5)	0.0008 (4)	0.0023 (4)
C22	0.0211 (5)	0.0324 (6)	0.0202 (5)	−0.0094 (4)	0.0017 (4)	0.0021 (4)
O22	0.0459 (5)	0.0322 (5)	0.0276 (4)	−0.0115 (4)	0.0041 (4)	−0.0029 (3)
O23	0.0375 (5)	0.0406 (5)	0.0188 (4)	−0.0053 (4)	0.0035 (3)	0.0053 (3)
C23	0.0387 (7)	0.0624 (9)	0.0191 (6)	−0.0070 (6)	0.0040 (5)	−0.0018 (5)

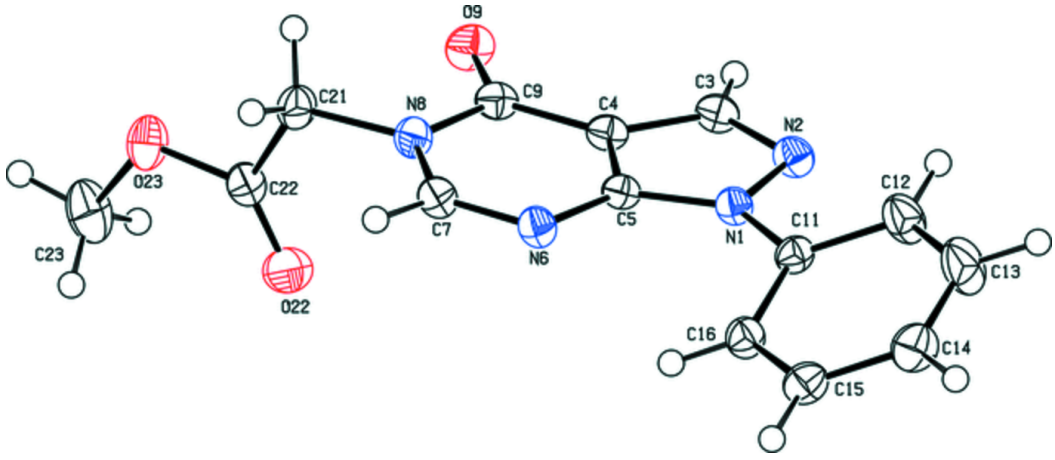
Geometric parameters (Å, °)

N1—C5	1.3672 (13)	C12—H12	0.9500
N1—N2	1.3838 (12)	C13—C14	1.3898 (19)
N1—C11	1.4311 (13)	C13—H13	0.9500
N2—C3	1.3286 (14)	C14—C15	1.3904 (17)
C3—C4	1.4148 (15)	C14—H14	0.9500
C3—H3	0.9500	C15—C16	1.3949 (15)
C4—C5	1.3933 (14)	C15—H15	0.9500
C4—C9	1.4379 (15)	C16—H16	0.9500
C5—N6	1.3745 (13)	C21—C22	1.5216 (16)
N6—C7	1.3052 (14)	C21—H21A	0.9900
C7—N8	1.3739 (14)	C21—H21B	0.9900
C7—H7	0.9500	C22—O22	1.2007 (15)
N8—C9	1.4253 (15)	C22—O23	1.3415 (13)
N8—C21	1.4629 (14)	O23—C23	1.4591 (16)
C9—O9	1.2274 (13)	C23—H23A	0.9800
C11—C12	1.3947 (16)	C23—H23B	0.9800
C11—C16	1.3956 (15)	C23—H23C	0.9800
C12—C13	1.3908 (17)		
C5—N1—N2	110.59 (8)	C14—C13—C12	120.96 (11)
C5—N1—C11	130.25 (9)	C14—C13—H13	119.5
N2—N1—C11	119.01 (8)	C12—C13—H13	119.5
C3—N2—N1	106.00 (9)	C13—C14—C15	119.36 (11)
N2—C3—C4	111.14 (9)	C13—C14—H14	120.3
N2—C3—H3	124.4	C15—C14—H14	120.3
C4—C3—H3	124.4	C14—C15—C16	120.59 (11)
C5—C4—C3	105.19 (9)	C14—C15—H15	119.7
C5—C4—C9	120.20 (10)	C16—C15—H15	119.7

C3—C4—C9	134.53 (10)	C15—C16—C11	119.38 (10)
N1—C5—N6	126.48 (9)	C15—C16—H16	120.3
N1—C5—C4	107.09 (9)	C11—C16—H16	120.3
N6—C5—C4	126.42 (10)	N8—C21—C22	111.45 (9)
C7—N6—C5	112.58 (9)	N8—C21—H21A	109.3
N6—C7—N8	126.36 (10)	C22—C21—H21A	109.3
N6—C7—H7	116.8	N8—C21—H21B	109.3
N8—C7—H7	116.8	C22—C21—H21B	109.3
C7—N8—C9	123.41 (9)	H21A—C21—H21B	108.0
C7—N8—C21	119.58 (10)	O22—C22—O23	125.04 (11)
C9—N8—C21	117.01 (9)	O22—C22—C21	125.21 (10)
O9—C9—N8	120.64 (10)	O23—C22—C21	109.73 (10)
O9—C9—C4	128.34 (11)	C22—O23—C23	114.78 (10)
N8—C9—C4	111.00 (9)	O23—C23—H23A	109.5
C12—C11—C16	120.46 (10)	O23—C23—H23B	109.5
C12—C11—N1	119.03 (10)	H23A—C23—H23B	109.5
C16—C11—N1	120.51 (9)	O23—C23—H23C	109.5
C13—C12—C11	119.24 (11)	H23A—C23—H23C	109.5
C13—C12—H12	120.4	H23B—C23—H23C	109.5
C11—C12—H12	120.4		

C5—N1—N2—C3	−0.39 (12)	C5—C4—C9—O9	178.62 (11)
C11—N1—N2—C3	−176.28 (9)	C3—C4—C9—O9	2.3 (2)
N1—N2—C3—C4	0.16 (12)	C5—C4—C9—N8	0.20 (14)
N2—C3—C4—C5	0.12 (12)	C3—C4—C9—N8	−176.09 (11)
N2—C3—C4—C9	176.79 (11)	C5—N1—C11—C12	−152.52 (12)
N2—N1—C5—N6	−178.17 (10)	N2—N1—C11—C12	22.44 (15)
C11—N1—C5—N6	−2.88 (18)	C5—N1—C11—C16	28.25 (17)
N2—N1—C5—C4	0.47 (12)	N2—N1—C11—C16	−156.79 (10)
C11—N1—C5—C4	175.76 (10)	C16—C11—C12—C13	−0.7 (2)
C3—C4—C5—N1	−0.35 (11)	N1—C11—C12—C13	−179.91 (12)
C9—C4—C5—N1	−177.61 (9)	C11—C12—C13—C14	−0.3 (2)
C3—C4—C5—N6	178.29 (10)	C12—C13—C14—C15	0.6 (2)
C9—C4—C5—N6	1.03 (16)	C13—C14—C15—C16	0.0 (2)
N1—C5—N6—C7	177.41 (10)	C14—C15—C16—C11	−0.90 (18)
C4—C5—N6—C7	−0.97 (16)	C12—C11—C16—C15	1.26 (17)
C5—N6—C7—N8	−0.33 (16)	N1—C11—C16—C15	−179.52 (10)
N6—C7—N8—C9	1.61 (18)	C7—N8—C21—C22	−101.82 (12)
N6—C7—N8—C21	−177.63 (11)	C9—N8—C21—C22	78.89 (12)
C7—N8—C9—O9	−179.96 (10)	N8—C21—C22—O22	12.05 (16)
C21—N8—C9—O9	−0.71 (15)	N8—C21—C22—O23	−169.38 (9)
C7—N8—C9—C4	−1.40 (14)	O22—C22—O23—C23	0.15 (17)
C21—N8—C9—C4	177.86 (9)	C21—C22—O23—C23	−178.43 (10)

Fig. 1


O9
C21
N8
C9
C4
C3
N2
O23
C22
C7
N6
C5
N1
C11
C12
C23
O22
C13
C16
C14
C15